FR02681138A1

Improvements with the chromatographic columns.

Chromatographic column consisting of a struc-3re support in which a structure of refillable the cartouche type is inseree, caracterisee by IE makes that the structure of support is constituee by modules supports or superposable casings of form generate cylindrical or prismatic into which are introduced likely modular cartridges d'etre empilees and whose IE garnishing can be cup by a common system d'au-tocompression of l'ensemble, the aforementioned cartouches being able to be traversees successively or separately by one or more fluids whose l'un ensures l'auto-compression.

IMPROVEMENTS WITH THE CHROMATOGRAPHIC COLUMNS The presente invention milked, has improvements with the chromatographic columns. Former L'art in this field is very abundant, but one will retain nevertheless the following documents consideres like caracteristiques. The French patent 2.556.099 (Industrial Group of Achievements and d7Applications) decrit a column of chromatography constitute by a tube of which a part contains a garnishing and comprising, has one of its ends, the means of communication with i^xterior, and d"autre share, at least a sliding body deplacant themselves longitudinally in the tube and presentant means of connection with a conduit communicating with Vexterieur, the aforementioned column etant caracterisee in what the aforementioned sliding body is deplace by the pressure exercee by the fluid under pressure inxecte in an enclosure formee between the surface of the sliding body opposee has ceile with the contact with the garnishing and I^xtremite of the tube which faces him. From this system on 1"auto-compress ion of the garnishing by the chromatographic fluid itself bases (fluid A chromatographier, eluant or solvent, primarily), are nes various improvements tending has to simplify necessary handling, inter alia A the installation and has I7extraction of the garnishing. One will quote in particular the French patent 2.606.882 (in the name of deposant of the presente dciaande) which decrit a column comprising, has its ends of the porous plates, of which I^une is fixed and fautre likely of deplacer has Vinterieur of the column under Inaction d^un jack and to thus compress the load contained in the aforementioned column which is caracterisee in what the load is contained in one 5 interchangeable cartridge rigid or semi-rigid d'un diameter exterior appreciably egal with the internal diameter of the column, this derniere etant coupee in two egaux half-cylinders that I^on brings closer I"un I^autre after insertion of the aforementioned cartridge and that I7 one holds greenhouses by any means together adapts. One thus could obtain columns has car-compression and has filling by cartouches what obliges nevertheless has to lay out, for each diameter of column of batteries of cartridges according to the necessary height of garnishing, c^est-a- to say according to the nature of the operations d'analyse, of separation,

d7extraction or other, according to the nature of the products has chromatographier and of the fluids use. If l'on does not want to invest in a series of columns, one can be thus amene, for example, has to work in a column of which the height corresponds to the maximum has to consider, leaves is ñ utiiiser qu^une cartridge height much more reduite in certain cases. The purpose of the presente invention is to return structures of columns and flexible cartridges, which makes it possible to make evoluer 1^ensemble with the gre needs, while keeping brackets time IE principle d^auto-compression by fluid IE has itself and that of the interchangeable cartridges.

According to 1^invention, one uses modules of columns constitute by modules of supports or casings of form R generate cylindrical or prismatic into which are introduced likely modular cartridges d/etre empilees and whose IE garnishing can be cup by autocompression of V together, the aforementioned cartouches which can 5 be traversees successively or separately by one or more fluids whose l^un ensures 1'auto-compression. In a same family of carrying modules, one can possibly inserer either cartridges of diameters exterieurs corresponding to the internal diameter of modules 10 carrier or the cylinder registered in IE case of the prismatic modules, or of the cartridges of diameters inferieurs thanks to the adequate shapes of the cartridges, or has spacers. In each cartridge. IE garnishing is contained between 15 two likely sliding elements extremes d^etre cross-pieces by the fluids, to ensure the chromatographic operations. For better rendering comprehensible the caracteristic techniques efc the advantages of the presente invention, one goes into 20 decrire an example of realization, etant of course that this one ñest not restrictive as for its mode of implementation and the qu^on applications can make some. One will refer has the single figure which represente schematiquement, out of partial longitudinal section, a chromatographic column 25 conforms has the presente invention.

L'ensemble includes/understands a basic part 1 montee on a support such q^un trepied, constitutes here by three feet independants 2 screw in base 1, which returns V together H dismountable and aisement transportable, as one IE will see d^illeurs throughout this description. On this basis 1 under the conditions decrites one or more modules of columns come to be fixed hereafter constitute 5 by carrying modules or casings such as 3 and of the also modular cartridges such as 13. On this base 1, comes to fix module support of cartridge 3, of form generate cylindrical or possibly prismatic A rolls registers, whose IE internal diameter corresponds to the diameter exterior of the cartridges which will come to be inseres there, with IE simple play necessary for the installation. It will be noticed however that in supports such as 3 of internal diameter such as defini-cidessus, it is possible d^inserer cartridges of diameters interieurs smaller than those corresponding has the family of cartouches s^inserant exactly in that of the carrying modules, since l^homme of l^art prevoit of the shapes of cartridges or spacers

ensuring IE maintenance in place of the cartridges in the carrying modules. The carrying module 3 can be goes up on basis 1 has faide d^un system has bayonet. Il presente of the openings or days lateraux on which one will return hereafter and which make it possible to facilitate the operations of motage and connection of the modular cartridges. Various supports such as 3 can be thus go up superimpose, consequently qu^ls presentent the same internal diameter, efc the means of fixing between them. It can be system has bayonet such that uses for the assembly on basis 1 of support 3. Il is preferable that each modular support presente a raccort male has one H end and female have I^autre, which simplifies I^nterchangeabilite, the assemblies and ensure has the time a good mecanic resistance and a good orientation of the modules, the carriers being used for the maintenance of I7ensemble of the 5 column, in particular during the phases of compression of the garnishing and the they-same chromatographic phases.

In base 1 a piston 4 is insere which presente a part of broad diameter 5 sliding in a alesage correspondent of base 1, prolongee towards IE low coaxialement by a stem 6 of smaller sliding diameter in an axial hole N correspondent of base 1 and crossing it. Traditional ^oits and other devices d^'etancheite are prevus between piston 4 and stem 6 d^ne share and the corresponding alesages and holes d^autre share. Piston 4 and the fcige 6 are borers axially to form a central conduit 7 that 1'on can close partly low by a stopper filete 8 or that Von can connect thanks to the threading of conduit 7 has any source of chromatographic fluid. Conduit 7 presente a radial derivation 9 which lead in a space ranging between base 1, piston 4 and the stem 6, and which includes/understands a part punt 10 in a plan of cross-section, and an annular part 11 between stem 6 and bases 1. The part punt 10 is constitute by a cavity of the base of diameter a little smaller than that of part 5 of piston 4 and L alesage correspondent of base 1, of gus kind I^paulement thus cree on the periphery of this part 10 empeche IE piston 4 to come in contact with the bottom from this cavity, and makes it possible the fiuide to be left again on all corresponding surface, thus ensuring the best conditions has 1"auto-compress ion. Towards high IE, IE piston 4 is prolonged coaxialement by a cylindrical part 12 of diameter inferior has that of the broadest part 5, left 12 destinee has to slide has soft friction in the cartridge 13 which will be to him superposee 5 per insertion in IE modulates support 3. The axial conduit 7 cross-piece also this part 12 and ends has 1/extremite higher 1^ensemble piston 4 (c/est-A-statement qu^il travirse 5, 6 and 12). Cartridge 13 is constitute by a segment of tube rigid or semi-rigid resistant the pressure of the garnishing in progress d^ operation has and forming a segment of the chromatographic column itself, whose useful IE diameter is the internal diameter of cartridge 13. The garnishing 14 is maintained in cartridge 13 between two sliding discs 15 and 16 presentant each one, of the external dimension, a full part rigid and sliding provided with edges exterieurs, respectively 17 and 18, dimension of the garnishing and percee axially, and internal dimension, c"est-A-statement, dimension of the garnishing, a porous disc maintained between edges 17 and

18 of the full discs rigid respective. This allows the fluid being able by conduit 7 of piston 4 to cross the disc full rigid 15 and to leave again themselves in the porous disc 17 to cross the garnishing 14 then. One can prevoir has the surface of the disc full rigid 15 with the radial grooves, preference, the dimension of the porous disc 17, which facilitates the distribution of the fluid in all the section of the porous disc 17 and the garnishing 14. The disc full rigid 15 can present, according to a mode of realisationprefere of 1/invention, an axial tubular prolongation 37 which comes F s^nserer in a alesage correspondent of the higher part D;u piston 4. Joints or other devices d^tancheite, are prevus by I^homme of Vart between disc full rigid 15 and cartridge 14, and between tubular part 37 5 of disc 15 and piston 4.

The disc full rigid 16 and IE porous disc 18 can be constitute same way that those which come d^etre decrits, including a tubular prolongation 38 to the top which penetre symetriquement in the piston 19 superior that I'on insere in cartridge 13 on disc 16. Its diameter single exterior is that of the high part 12 of piston 4, c^est-A-statement qu"il can slide has soft friction in cartridge 13. Il presente an axial conduit 20 on a part its height, dimension presentant the court alesage in which: penetre the tubular part 38 of the disc full rigid 16. A radial derivation 21 percee in this piston 19 puts in communication leads it 20 with exterior thanks to a connection filete, this which-allows the connections on which one will return further. The piston 19 presente moreover of the epaulements or butees exterieures 39 which empechent the cartridge to assemble too high Durand the operations, in particular during 1"auto-compression, which would be likely to make echapper the disc full rigid 15 and the garnishing qu7!! maintains. The module support 3 presente of the openings laterales 22 and 23 which allow d'acceder aa connection of conduit 21 and maitriser and verifier the correct installation of the elements, such as for example that of piston 19 in cartridge 13 on the disc full rigid 16. On IE support 3 east modulates assembles way equivalent has what comes d^etre decrit a second carrying module 3', a piston 19 /, a cartridge 13/with its rigid garnishing 14 /, its two full discs 15' and 16 /, its two porous discs 17r 5 and 18", and its pistons 19' and 19". On the figure, one A defers the same references on the same elements by marking them in way such as 19 and 19" to indicate qu^il s^git d7elements equivalents of I7element 19. One will not decrira these elements in detail, consequently, for example, that the assemblies of the four pistons with the corresponding discs and cartouches are equivalents. The full and rigid discs can present, for example of the radial grooves, like it has ete said in connection with first disc 15 of the dimension or they are in contact with the porous discs. On IE modulates support 3', one can go up, always thanks to a system of preference has bayonet, a hat 28 in which is montee axially a screw 27 has tete moletee destinee has to come s^appuyer on IE piston superior 16' who presente, of preference like all the pistons (in particular superieurs such as 16) and to facilitate Vinterchangeabilite, a central alesage 26 low height in which s"appuyer comes screw 27. The d7utilisation mode and of operation of this unit is as follows: One

assembles base 1 on his trepied 2. One installs piston 4, stem 6 beam base 1 and appearing below. The bottom of the broad part 5 of piston 4 must come to rest in butee on the bottom of 1' alesage correspondent of base 1, by menageant I^espace 10 there.

R One assembles IE modulates support 3 on basis 1 then one y insere cartridge 13 while I^enfoncant in order to bring in contact with piston 4, IE disc full rigid 15. One insere IE piston 19 in the cartridge 13 in order to 5 1/to bring in contact with the disc full rigid 16. One verifie IE correct assembly of this whole in way which the circulation of the fluid can s^effectuer without escape. One verifie also that IE connection of conduit 21 is in position accessible by Vune from the openings 22 or 23 of the module support 3. If Von desire to work with two modular sets, one sets up IE second module support 3 on IE first module support 3, then IE third piston 19 in IE second module 3 /, the second cartridge 13 in IE second module support 3' , and IE fourth piston 19" in IE second module support 3' and the second cartridge 13/. One verifie contacts between IE third piston 19 and d7!^^ share IE second piston 19 and d^utre share. IE full disc rigid 15f, thus qu^entre IE fourth piston 19" and IE disc full rigid 16/. One verifie also 1'" accessibility with the connections of conduits 21 and 21". One sets up IE hat 28 on IE second module support 3/. One verifie IE assembly of the systems has bayonets and one tightens without excesses screw 27, which ensures IE maintenance in place and with the various contacts of If together which comes d^tre decrit. One connects IE led 7 A the source of fluid, the connections of conduits 21 and 21 /, which assembles in series two cartridges 13 and 13", and one connects IE led 21" towards a receipt or an unspecified evacuation. The drains of connection are not representees on the figure. R One leaves fluid IE penetrer under pressure in IE leads 7 and led IE of derivation 9. Because of the pressure losses in the cartridges. Fluid IE starts by pushing 1'ensemble pistons discs and garnishings has meeting of the screw 27 5 being used as butee. When the pressure in I^espace 10, 11 comes equilibrer that in the garnishings, taking into account the pressure losses in the garnishings and of the differences in surfaces between that of part 5 of the piston 4 and that lower of part 12, corresponding A the finished cross-section of the column, 1"auto-compression is assuree and 1" chromatographic operation itself deroule. The fluid crosses IE led 7, IE tubes 37, IE disc full rigid 15, the porous disc 17, the garnishing 14, the porous disc 18, the disc full rigid 16, conduits 20 and 21, the drain of connection not representee on the figure, conduits 21 and 20 /, the full disc rigid 15' , the porous disc 17 /, the garnishing 14 /, the porous disc 18 /, the disc full rigid 16 and conduits 20 and 21/. One will notice inter alia: - that I7auto-compression acts on I7ensemble of the cartridges, independamment of connections; that the modular height between balonnettes of the modules supports is, of preference, appreciably of the same order that the height of the corresponding cartridges provided with the pistons; - that the cartridges can be montees in series, in parallele or independamment/ what can allow several operations on several different fluids, and/or with different garnishings; ) R that I^on can modulate the total height of the

garnishings according to the needs, while having of some modules supports and some cartridges; for example for a useful diameter of 50 A 100 mm, one can prevoir useful heights 5 of 100, 200, 300 and 500 mm; that I7on can use an unspecified number of Ca rtouches has to only count d^une and single; - that I^on can aisement reload the cartridges.

CLAIMS 1 chromatographic Column constituee by a structure support in which a structure of refillable the cartouche type is inseree, caracterisee by IE makes that the 5 structure of support is constituee by modules supports or casings superposable (3, 3") of form generate cylindrical or prismatic into which are introduced modular cartridges (13, 13/) likely d'etre empilees and of which IE garnishing (14, 14") can be cup by a common system 10 d^utocompression (4, 5, 6, 7, 9, 10, 11, 27, 28) of I"ensemble, the aforementioned cartouches being able to be traversees successively or separately by one or more fluids whose I^un ensures 1'auto-compress ion. 2 Column according to claim 1 caracterisee by IE makes that in modules carrying (3,3/) same internal diameter, are inseres cartridges (13, 13/) of diameters exterieurs inferieurs has that corresponding to the internal diameter of the modules carrying or the cylinder registered in IE case the prismatic modules, thanks to spacers. 3 Column according to I^une of claims 1 or 2 caracterisee by IE makes that, in each cartridge (13, 13/), IE garnishing is contained between two sliding elements extremes (4, 5, 6, 15, 16, 17, 18, 19, 19 /, 15 /, 16 /, 17 /, 18 and 19") likely d^etre cross-pieces by the fluids, to ensure the chromatographic operations. 4 Column according to I^ne of claims 1 has 3 caracterise by made IE that the sliding elements are constitute by interchangeable pistons (4, 19, 19 /, 19") places with contact of hard disks (15, 16, 15 /, 16/) of maintenance of the garnishings (14, 14/) in the cartridges (14, 14/). 5 Column according to claim 4 caracterisee by IE makes that the pistons presentent conduits (7, 20, 21, 21 /, 20 /, 5 20", 21") of connection at least a source of fluid has, of connection of the cartridges between them and d^evacuation. 6 Column according to I7une of claims 4 or 5 caracterisee by IE makes that center each disc full rigid (15, 16, 15 /, 16/) east goes up, of the dimension of the garnishing (14, 14/), one 10 porous disc (17, 18, 17 /, 18/). 7 Column according to I7une of claims 1 has 6 caracterisee by made IE that each element support (3, 3/) presente has an end a cabling system male and has I^autre end a system corresponding female, a base (1) and one 15 hat (28) presentant the corresponding systems for the fixing of the base and tete of the modules of column. 8 Column according to claim 7 carcterisee by IE makes that IE hat (28) presente a butee reglable (27) A Vencontre whose IE system d^auto-compress ion acts (4, 20 5, 6, 7, 9, 10, 11). 9 Column according to 1'une of claims I has 8 caracterise by made IE that the modules supports (3, 3') openings laterales presentent (22, 23, 22 /, 23/) d^acces and of control. 25